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(54) Title: COMPOSITIONS

(57) Abstract: There is described a novel fuel additive, novel fuel compositions and to a method to maximise fuel efficiency within existing infrastructures, without changing the characteristics of the engine. A fuel additive comprising an alkoxyated alcohol, an alkanolamide and an alkoxyated fatty acid characterised in that the alkoxyated alcohol has a molecular weight of greater than 281 and the fatty acid (without ethoxylation) is a C<sub>16</sub> to C<sub>30</sub> fatty acid. There is also described fuel compositions comprising the aforementioned additives.

### Compositions

This invention relates to a novel fuel additive, novel fuel compositions and to a method to maximise fuel efficiency within existing infrastructures, without changing  
5 the characteristics of the engine.

Many additive packages for fuel have tried to formulate a better conditioned fuel, most are impractical, as, *inter alia*, the cost of the dose required is greater than the benefit achieved.

10

British Patent number 2217229 describes a composition which uses 5-6% surfactant for 1% of water contamination. However, emissions are not addressed.

International patent application WO 98/17745 shows a more practical formula but  
15 uses relatively low molecular weight ethoxylated alcohols, such as Neodol 91/2.5 which has a molecular weight of approximately 281.

International Patent Application No WO 99/20715 addresses a similar type of process by isothermally mixing surfactants. It is described that the preferred  
20 ethoxylated alcohol has an average molecular weight below 200 or below 160, although ethoxylated alcohols such as Neodol 1/3 are specifically mentioned, which have a molecular weight of approximately 305. Furthermore for heavy fuels, such as diesel or Kerosene, the specification describes that higher molecular weight alcohols are required, such as Neodol 1/6 (molecular weight 437). Furthermore, the fatty  
25 acids referred to in the description are limited to C9 to C15 fatty acids.

A requirement in the commercial market place is for cleaner burning fuels, delivered at a comparable price and additised at a suitable dose to allow use immediately, without encompassing major test programmes and legislative changes. This invention  
30 seeks to deliver an additive package that achieves this and eliminates the requirement for thermal blending.

We have now surprisingly found novel fuel additives and novel fuel compositions made therefrom which improve on prior art compositions.

Thus according to a first feature of the invention we provide a fuel additive  
5 comprising an alkoxyated alcohol, an alkanolamide and an alkoxyated fatty acid, or an ester thereof, characterised in that the ethoxyated alcohol has a molecular weight of greater than 281 and the fatty acid (without ethoxylation) is a C<sub>16</sub> to C<sub>30</sub> fatty acid.

The alkoxyated alcohol is preferably an ethoxyated alcohol. It is essential that the  
10 ethoxyated alcohol is an oil soluble alcohol. Therefore, alkanols are preferred and these may be primary, secondary or tertiary alkanols and especially primary alkanols.

The degree of ethoxylation is chosen to optimise performances in the blend with the other two selected surfactants and may be from 1 to 20, but more preferably from 5 to  
15 12. A suitable product within this range would be, for example, that derived from the addition of 7 molecules of ethylene oxide to 1 mole of oleic acid, or a derivative thereof.

Preferably, the alkoxyated alcohol is an ethoxyated alcohol. The alkoxyated  
20 alcohol may have a molecular weight of greater than 281. Thus according to a preferred embodiment of the invention the alkoxyated alcohol has a maximum molecular weight of 790. Thus we provide a derivative as hereinbefore described characterised in that the alkoxyated alcohol has a molecular weight of from 305 to 790. Other molecular weight ranges which may be mentioned are from 305 to 569  
25 and from 305 to 524.

The Neodol alcohol ethoxylates are preferred. Ethoxylated Neodol 91 is especially preferred. Neodol 91 is a mixture of C<sub>9</sub> to C<sub>11</sub> alcohols and has an average molecular weight of 160. The ethoxylated Neodol alcohols are usually identified by the degree  
30 of ethoxylation, thus Neodol 91/2.5 has 2.5 degrees of ethoxylation per mole of alcohol. According to the invention the alcohol ethoxylate used may optionally

comprise a mixture of alcohol ethoxylates, e.g. a mixture of Neodols. Thus the alcohol ethoxylate may be selected from one or more of Neodol 91-6, Neodol 91-8, Neodol 1-9, Neodol 23-3, Neodol 23-5, Neodol 23-6.5, Neodol 25 and Neodol 45.

- 5 Specific Neodol alcohol ethoxylates which may be mentioned include one or more of Neodol 91-6, Neodol 91-8, Neodol 1-3, Neodol 1-5, Neodol 1-7, Neodol 1-9, Neodol 23-3, Neodol 23-5, Neodol 23-6.5, Neodol 25-3, Neodol 25-7, Neodol 25-9, Neodol 25-12, Neodol 45-2.25, Neodol 45-7 and Neodol 45-13. Especially preferred alcohol ethoxylates are Neodol 1-3, Neodol 1-6 and Neodol 1-8. Neodol alcohol ethoxylates  
10 are available from Shell Chemicals Limited in the UK.

Preferably the fatty acid is a C<sub>16</sub> to C<sub>20</sub> fatty acid, more preferably a C<sub>16</sub> to C<sub>18</sub> fatty acid and especially a C<sub>18</sub> fatty acid, e.g. oleic acid, or an ester thereof.

- 15 The oleic acid ethoxylate may comprise the free acid, an ester, a mixture of esters or a mixture of the acid and one or more esters. When an oleate ester ethoxylate is used, the ester may be an alkyl oleate, preferably a C<sub>1</sub> to C<sub>10</sub> alkyl oleate, such as ethyl oleate and especially methyl oleate.
- 20 The alkanolamide is preferably an ethanolamide and more preferably a diethanolamide. Especially preferred are the diethanolamides and particularly the super diethanolamides. By the term super diethanolamide we mean a diethanolamide in which the nitrogen is substituted by an acyl substituent e.g. acyl C<sub>5</sub> to C<sub>20</sub>, preferably C<sub>8</sub> to C<sub>18</sub>, more preferably C<sub>10</sub> to C<sub>18</sub>. The most preferred diethanolamide  
25 has a C<sub>18</sub> acyl substituent i.e. oleic diethanolamide.

There are three commercial routes to alkanolamides;

- Acid + alkanolamine = alkanolamide + water
- 30 Plant or animal oil (triglyceride) + alkanolamine = alkanolamide + glycerol
- Methyl ester + alkanolamine = alkanolamide + methanol

These are listed in order of increasing product quality. The route via the acid often uses an excess of alkanolamine to produce a product higher in amide than is obtainable from the acid if a stoichiometric ratio is used; these products are sometimes referred to as Kritchevsky amides. The products derived from reaction of substantially stoichiometric proportions of an alkanolamide with a fatty acid ester, typically a methyl or glyceryl ester, are referred to as super amides. Generally the alkanolamides may be prepared by conventionally known methods, such as, those described by Shelmerdine et al "Industrial Applications of Surfactants II".

10

The preferred diethanolamide would have a molecular weight of around 380, e.g. oleic diethanolamide.

With heavier fuels, such as diesel or kerosene ethoxylated alcohols with a molecular weight of from 281 to 790 may also be used in conjunction with higher molecular weight ethoxylated fatty acids, e.g. the C<sub>16</sub> to C<sub>30</sub> fatty acids and esters thereof. Although we have also found that with lower molecular weight fatty acids, or an ester thereof, e.g. C<sub>7</sub> to C<sub>15</sub>, and an alcohol with a molecular weight of less than Neodol 1/6 (MW 437) may be used, e.g. alcohol ethoxylates with a molecular weight of between 200 and 436 (e.g. Neodol 1/3), preferably between 281 and 436.

20

Specific Neodol alcohol ethoxylates which may be mentioned include one or more of Neodol 91-6, Neodol 1-3, Neodol 1-5, Neodol 23-3, Neodol 23-5, Neodol 25-3 and Neodol 45-2.25.

25

An additive of the invention may be added to a hydrocarbon fuel, e.g. diesel, petrol or alcohol, such as ethanol which may or may not be contaminated with water. Alternatively the hydrocarbon fuel may be a blend of a petroleum based fuel such as diesel or petrol, with an alcohol such as ethanol. The invention is seen to particularly good effect when added to synthetic fuels based on low fraction oils.

30

Thus, according to a further feature of the invention we provide a fuel composition comprising a hydrocarbon fuel and a fuel additive as hereinbefore described.

5 The fuel composition may be a gasoline, diesel (including biodiesel), gasohol (a mixture of gasoline and alcohol), oxydiesel (a mixture of diesel and an alcohol) or any of the aforementioned fuels with water present.

The concentration of the additive can be very low, typically of the order of 0.5 – 1:1200, preferably about 1:1200, most preferably 1:1000. There appears to be no  
10 technical or economic benefit in adding more unless a co-solvent dual action is required, when the priority will be dosage against performance.

In the prior art of WO 99/20715 it is preferred that higher molecular weight alcohol ethoxylates are used with diesel and kerosene fuels, for example, Neodol 1/6 and  
15 Neodol 1/8. Thus it is a novel feature of the present invention to provide a diesel or kerosene fuel which comprises diesel or kerosene and a fuel additive wherein the molecular weight of the alcohol ethoxylate is between 200 and 430 and the fatty acid is a C7 to C20 fatty acid

20 Thus, according to a further feature of the invention we provide a diesel fuel composition comprising diesel and a fuel additive comprising a diethanolamide, an ethoxylated fatty acid/ester and an ethoxylated alcohol, characterised in that the ethoxylated alcohol has a molecular weight of between 200 and 430.

25 Ethoxylated alcohols which may be used in the diesel fuel composition of the invention include Neodol 91/3.

The invention has various blends for different applications but assuming a mixture, the ratios of which are similar to that described in the prior art of WO 98/17745  
30 namely;

50% v/v alcohol ethoxylate  
25% v/v diethanolamide  
25% v/v fatty acid ethoxylate

5 (all thermally mixed at 55 – 58deg.C)

Thus a preferred composition of the present invention may be described as an additive mixture comprising;

10 20-80% v/v alcohol ethoxylate  
10-40% v/v diethanolamide  
10-40% v/v fatty acid ethoxylate.

15 An especially preferred composition of the present invention may be described as an additive mixture comprising;

30-70% v/v alcohol ethoxylate  
15-35% v/v diethanolamide  
15-35% v/v fatty acid ethoxylate.

20

The composition may preferentially be blended at room temperature.

25 Preferably the ethoxylate of the fatty acid makes-up about 25% by volume of the additive and further preferably the alcohol ethoxylate comprises 50% by volume of the additive.

In another aspect the invention provides a fuel composition comprising a light weight fraction and a surfactant fuel additive as hereinbefore described.

30 In the diesel composition of the invention in which the fatty acid is not limited to a C16 to C20 fatty acid, the fatty acid ethoxylate may comprise any conventionally

known fatty acid ethoxylate or a derivative thereof. Thus the fatty acid ethoxylate may be derived from a fatty acid having from 7 to 20 carbon atoms, preferably from 10 to 18. The fatty acid may be a saturated or unsaturated fat. By the term "alkoxylated fatty acid or a derivative thereof we mean a derivative of the acid, for  
5 example, an ester, e.g. an alkyl ester. The most preferred fatty acid is an unsaturated fatty acid and especially C<sub>18</sub>, oleic acid or a derivative thereof, such as an oleate ester, e.g. an alkyl C<sub>1</sub> to C<sub>10</sub> oleate. Derivatives which may be mentioned include an ethyl oleate or a methyl oleate. In one embodiment of the invention, when ethanol is present then the fatty acid is greater than C<sub>15</sub> and especially oleic.

10

The benefit of using an oleate over and above an ethoxylated C<sub>14</sub> fatty acid is; an oleate has a double bond which in certain surfactant chemistry could be a problem, but is in this invention a considered bonus, due to its composite relationship flexibility. The preferred molecular weight being above 500 for the oleate.

15

The relationship of the oleate with the correct molecular weight alcohol ethoxylate and the ability to produce this formula at room temperature allows for integration into the existing fuel infrastructure, unlike any previous art.

20 A programme in the USA has launched a 15% blend of ethanol with regular diesel and used between 1 and 5% additive to achieve this. The result of this blend is a loss of fuel consumption and legislation to accept the additive as a fuel and not an additive.

25 The USA requirement for fuel ethanol to be blended in a diesel/ethanol mixture is 95% proof ethanol. By adding our formula, utilising the benefit of the reduced dose due to the oleate, we are able to treat the ethanol with 5% by volume of our formula and keep within specification for fuel ethanol.

30 A preferred fuel composition of the invention is a blend of regular diesel, an oxygenator, e.g. ethanol, and an additive as hereinbefore described.



The use of a surfactant composition comprising a fatty acid ethoxylate or a derivative thereof is especially advantageous in conjunction with diesel fuel compositions and especially diesel/alcohol compositions. Thus, according to a further feature of the invention we provide a fuel composition comprising a diesel fuel, an alcohol and a  
5 surfactant additive as hereinbefore described.

The alcohol is preferably ethanol. Optionally, the diesel composition of the invention may additionally include the use of an alkyl ester of oleic acid and a co-solvent of an alkyl alcohol, e.g. a C<sub>3</sub> to C<sub>6</sub> alcohol.  
10

Ethanol present may be up to at least 10%v/v ethanol in the fuel or above 18%v/v.

Attempts have been made to blend ethanol into diesel and achieve certain benefits expected as per gasohol, a long standing cleaner burning fuel.  
15

We also provide a method of running an engine adapted to use a alcohol-based fuel, comprising adding to the fuel a miscible additive selected to solubilise the fuel and the additive so eliminating the deposit of by-products formed during the combustion of the fuel.  
20

Thousands of low dose additives are available on the market making claims that they reduce emissions or increase miles per gallon. These additives range from alcohol octane boosters to detergents. One thing is common, they never claim to reduce CO<sub>2</sub>.

25 Our additive has a treatment ratio of 1.1000 and 1.500 for gasoline and diesel respectfully. By understanding that in the modern day motor vehicle emissions are so low that if you were able to reduce CO and HC to zero emissions the claims for an increased miles per gallon number would be considered within noise, i.e. less than 3%. Using the carbon balance theory i.e.. The carbon in the fuel returns through the  
30 exhaust after combustion as CO, HC or CO<sub>2</sub> it is impossible to claim more mpg without reducing CO<sub>2</sub>.

CO<sub>2</sub> is known as a harmless gas but recently has been associated with global warming. Another way of increasing mpg is utilising less weight in the vehicle or reducing the engine size from 2 litre to 1.6 litre for example. Direct injection is another way to reduce CO<sub>2</sub>.

5

Our invention reduces CO, HC, NO<sub>x</sub> and CO<sub>2</sub> without changing the inertia weight or engine displacement of the vehicle at a treatment rate of 1.1000 for gasoline and 1.500 for diesel. With diesel black smoke opacity and particulates are also reduced. Prior art suggests that it is not possible to reduce NO<sub>x</sub> and particulates at the same

10 time.

It is the precise aim of the formula to create the best solubilisation of the additive into the fuel, this will then reduce the resistance in the fuel pipeline by creating a permanently repairable monolayer, which in turn optimises vaporisation of the fuel.

15

E 85 Alternative fuel. A blend of 85% ethanol and 15% gasoline

E85 is being sold as an alternative vehicle fuel. The fuel conforms to EPACT and 20% of fleet owners in US have to use alternative fuel by 2000.

20

This invention at very low dose 1.1000 reduces emissions and eliminates cold start problems caused by condensation and water contamination which in turn causes phase separation.

25 At cold start fuel separation gives invariable and inconsistent RVP. making it extremely difficult for spark ignition starting. As 85% of the fuel is ethanol which is hygroscopic water contamination occurs and the hydrocarbon separates from the alcohol. As cold start is difficult, the fuel with water will find entry into the sump and cause emulsification of the engine oil and any alcohol there will cause degradation

30 during cold start running.

Another form of degradation is caused by per acid reacting with per oxides caused by the increase in temperature of the returning unused fuel which in turn causes condensation. These reactions are known to create iron formate in the piston chambers and not iron oxide as was previously thought. Iron formate is extremely  
5 corrosive compared to iron oxide.

This invention seeks to eradicate the inconsistencies of cold start and eliminate the emulsifying of engine oil at cold running complete with suppressing the peroxide reaction therefore minimising engine degradation.  
10

A more complete burn will decrease the aldehyde emissions expected from alcohol fuels. The ability to eliminate cold start and oil emulsification is characteristic of the formula applied to the 1.1000 additive.

15 The invention will now be described by way of example only.

#### **Example 1a**

An additive of the invention was prepared by blending:

20

Neodol 91-6	1 litre
diethanolamide	500ml
polyoxyethene-7-oleate	500ml

#### **25 Example 1b**

Example 1a was repeated using Neodol 91-8.

#### **Example 1c**

30

Example 1a was repeated using Neodol 1-3.

**Example 1d**

Example 1a was repeated using Neodol 1-5.

**5 Example 1e**

Example 1a was repeated using Neodol 1-7.

**Example 1f**

10

Example 1a was repeated using Neodol 1-9.

**Example 1g**

15 Example 1a was repeated using Neodol 23-3.

**Example 1h**

Example 1a was repeated using Neodol 23-5.

20

**Example 1i**

Example 1a was repeated using Neodol 23-6.5.

**25 Example 1j**

Example 1a was repeated using Neodol 25-3.

**Example 1k**

30

Example 1a was repeated using Neodol 25-7.

**Example 1l**

Example 1a was repeated using Neodol 25-9.

**5 Example 1m**

Example 1a was repeated using Neodol 25-12.

**Example 1n**

10

Example 1a was repeated using Neodol 45-2.25.

**Example 1o**

15 Example 1a was repeated using Neodol 45-7.

**Example 1p**

Example 1a was repeated using Neodol 45-13.

20

**Example 2**

We applied 7.7% ethanol treated at 5% by volume with our formula and achieved the following.

25

Cetane 53.9

Base diesel. 53.4 CARB approved diesel.

Oxydiesel. 53.9 consisting 92.3% CARB approved diesel and 7.7% ethanol which

30 was treated with our formula and Cetane improver totalling 0.385% of the total fuel.

		Base diesel deg.F.	Oxydiesel deg.F.
	IBP	368.7	170.6
5	T5	405.5	173.3
	T10	425.5	381.6
	T20	452.5	434.3
	T30	475.0	460.2
	T40	496.6	482.4
10	T50	517.6	503.4
	T60	539.2	526.3
	T70	562.6	550.8
	T80	588.5	578.1
	T90	624.5	613.0
15	T95	658.6	646.9
	FBP	671.5	665.6
	Recovery	97.8 vol.%	98.3 vol.%

20

This showed a reduction in distillation temperatures, all through the curve, giving a denser charge at the point of combustion, which in turn generates a cleaner burn. The other unique characteristic is the ability to change the initial boiling point of the diesel. T10 is 381.6 for Oxydiesel and 425.5 for CARB diesel. As 7.7% of the fuel is not CARB diesel and 10% by volume of the fuel has boiled off at T10 the distillation curve shows a predicted more complete burn over a longer period of time, starting earlier with the diesel.

25

By lowering T50 a NO<sub>x</sub> reduction was achieved.

30

Finally the extra percentage recovered 0.5% provides increased efficiency.

**Example 3**

Gasohol. (90% gasoline, 10% ethanol)

5

Since the mid-seventies certain states in USA have opted to oxygenate their gasoline with 10% ethanol. As concerns have shifted towards evaporative emissions Reid vapour pressure has been a prominent test checking for evaporative emissions. This is in accordance with ASTM test D5191.

10

Ethanol, which, alone has an RVP of less than 3, when blended with gasoline increases the RVP of the host fuel by at least one whole number over and above the gasoline RVP.

15 These volatile evaporative emissions have created problems in California, USA. In California the maximum RVP for gasohol is 7, with ethanol as an oxygenate then the gasoline must be blended at a maximum of 6 RVP to accommodate the ethanol problem. By blending the composition of the invention into ethanol at 0.5% of the fuel, the RVP is suppressed sufficiently and refiners can blend 6.9 RVP gasoline for  
20 gasohol, cheapening the product cost considerably and performing legally within the California Air Resources Board (CARB) legislation.

Thus according to a further feature of the invention we provide a composition as hereinbefore described with an RVP of 7 or less.

25

Furthermore, alcohol will increase the octane of fuel by up to 3 whole numbers. The middle octane blend in California is a mix of high and low octane fuel, with the aid of our formula a blender can gain advantage by utilising formula treated ethanol as part of this process reducing the cost of blends keeping the RVP within the required  
30 limits.

**Example 4****Aqueous diesel**

For some time aqueous diesel has been known to reduce NO<sub>x</sub> from exhaust emissions  
5 NO<sub>x</sub> contributing towards smog etc. Although it is an achievement to reduce NO<sub>x</sub> by  
adding water to the fuel the calorific loss shows a substantially less viable alternative.

We tested 7% water with less than 7% additive from the formula below and achieved  
comparable NO<sub>x</sub> reductions to any prior art. However this was blended at room  
10 temperature and a clear stable homogenous fuel was formed which could be totally  
integrated into the existing infrastructure.

**Formula:**

15 40-60% ethoxylated alcohol with a molecular wt. above 281  
20-30% ethanolamide with a molecular wt. around 250  
20-30% polyoxyethylene-7-oleate with a molecular wt. in excess of 500  
blended at room temperature.

**20 Example 5**

Tests prove that easier cold starting and more mpg coupled with the reduction in  
aldehydes will give an overall performance increase of the vehicle.

**25 Formula:**

40-60% ethoxylated alcohol mol.wt over 281  
20-30% ethanolamide mol.wt over 250  
20-30% polyoxyethylene7-oleate mol.wt. over 500  
30 all blended at room temperature.



**Example 6**

oil soluble primary alcohol ethoxylate. An additive was  
5 prepared according to Examples 1 to 5 available as NEODOL 1 litre  
diethanolamide 500 ml  
a fatty acid or an ester thereof 500 ml

Different used vehicles, having Diesel and petrol engines, were tested at a local  
10 Ministry of Transport test house. The fuel tank of each was filled, and the vehicle  
driven for about 112 Km at an average speed of 96 Kph. A dose of the stock solution  
was added to the tank of each vehicle in a volume ratio of 1:1000. Visual inspection  
showed that a clear homogeneous solution was formed. The tank was refilled and the  
vehicle then driven again over the same journey. The MOT test was repeated.

15

The results showed a decrease in fuel consumption.

The tests showed the following reductions in emissions:

20

Petrol Engine

CO reduced

Hydrocarbon reduced

Diesel Engine

Diesel smoke reduced

25

**Example 7**

A Mercedes M111 basic test engine was cleaned and prepared for testing to record  
any changes in reference gasoline without additive and with additive at a treatment  
30 rate of 1:1000.

The standard methods of measurement were used in accordance with NAMAS specifications, particular interest was paid to LAMBDA as the leaning/richening of the engine would not encourage comparable results. LAMBDA was set At  $\lambda=0.05$

- 5 The basic test was started and the engine was run hot and then dropped from 4,500 r.p.m. WOT to 1,800 r.p.m. PT stopping at different conditions to enable comparisons. LAMBDA performed at  $\lambda=0.05$ . At the end of the first test the head was cleaned and once again the test was repeated with additive at 1:1,000.

10 **Example 8**

- A Bench Test was carried out under controlled laboratory conditions to ascertain Fuel Consumption and Emission Performance at 1,800 r.p.m. and 2,500 r.p.m. part throttle and also measuring Power Curve and Torque Curve Performance, using RF83  
15 reference European non-additised fuel, with all measurements recorded to NAMAS Criteria. The engine was a MERCEDES 2 litre M111 Bench Engine suitable for unleaded fuel, fitted with a Catalytic Converter.

**Example 9**

20

A test was carried out to measure any reduction in  $\text{NO}_x$  as  $\text{NO}_x$  is directly related to combustionability and is a hazard that is impossible to negate in engines as Air/Fuel Ratio will always contain Nitrogen. The results showed that  $\text{NO}_x$  reduced.

25 **Example 10**

- A specific variety of fuels from premium grade gasoline, industry standard diesel and various alcohol blended fuels were selected and from each 100 ml were transferred to each of twelve 200 ml measuring cylinders for reference to the phase separation  
30 caused by saturation of water to the polymer. The optimal being two titrations previous to the phase.

After the introduction of each titration the solution was gently stirred for twenty seconds. The resultant effect was left for ten minutes to settle before visible results were recorded.

## 5 Example 10a

Fuel	No	Water Content	Additive
Gasoline	1	0%	0%
Gasoline	2	10%	0%
Gasoline	3	10%	10%
Gasoline	4	10%	9%
Gasoline	5	10%	8%
Gasoline	6	10%	7%
Gasoline	7	10%	6%
Gasoline	8	10%	5%
Gasoline	9	10%	4%
Gasoline	10	10%	3%
Gasoline	11	10%	2%
Gasoline	12	10%	1%

10 After the introduction of each titration the solution was gently stirred for twenty seconds. The resultant effect was left for ten minutes to settle before visible results were recorded.

## Example 10b - Gasohol

Consisting of 90% regular unleaded gasoline with 10% denatured ethanol

Fuel	No	Water Content	Additive
Gasohol	1	0%	0%
Gasohol	2	10%	0%
Gasohol	3	10%	10%
Gasohol	4	10%	9%

Gasohol	5	10%	8%
Gasohol	6	10%	7%
Gasohol	7	10%	6%
Gasohol	8	10%	5%
Gasohol	9	10%	4%
Gasohol	10	10%	3%
Gasohol	11	10%	2%
Gasohol	12	10%	1%

**Example 10c – Diesel**

Fuel	No	Water Content	Additive
Diesel	1	0%	0%
Diesel	2	10%	0%
Diesel	3	10%	10%
Diesel	4	10%	9%
Diesel	5	10%	8%
Diesel	6	10%	7%
Diesel	7	10%	6%
Diesel	8	10%	5%
Diesel	9	10%	4%
Diesel	10	10%	3%
Diesel	11	10%	2%
Diesel	12	10%	1%

**5 Example 10 d - Alternative Gasoline (AH. Gas.)**

Consisting of alcohol and a blend of hydro carbons the majority percentage being alcohol

Fuel	No	Water Content	Additive
Gasohol	1	0%	0%
Gasohol	2	10%	0%

Gasohol	3	10%	10%
Gasohol	4	10%	9%
Gasohol	5	10%	8%
Gasohol	6	10%	7%
Gasohol	7	10%	6%
Gasohol	8	10%	5%
Gasohol	9	10%	4%
Gasohol	10	10%	3%
Gasohol	11	10%	2%
Gasohol	12	10%	1%

**Example 10e – Alternative Gasoline**

<b>Fuel</b>	<b>No</b>	<b>Water Content</b>	<b>Additive</b>
Alt Gas	1	0%	0%
Alt Gas	2	10%	0%
Alt Gas	3	10%	10%
Alt Gas	4	10%	9%
Alt Gas	5	10%	8%
Alt Gas	6	10%	7%
Alt Gas	7	10%	6%
Alt Gas	8	10%	5%
Alt Gas	9	10%	4%
Alt Gas	10	10%	3%
Alt Gas	11	10%	2%
Alt Gas	12	10%	1%

**5 Example 10f**

<b>Fuel</b>	<b>No</b>	<b>Water Content</b>	<b>Additive</b>
Gasoline	1	0%	0 %

Gasoline	2	5%	0 %
Gasoline	3	5%	5 %
Gasoline	4	5%	4.5%
Gasoline	5	5%	4 %
Gasoline	6	5%	3.5%
Gasoline	7	5%	3 %
Gasoline	8	5%	2.5%
Gasoline	9	5%	2 %
Gasoline	10	5%	1.5%
Gasoline	11	10%	1 %
Gasoline	12	10%	0.5%

**Example 10g - Gasohol**

<b>Fuel</b>	<b>No</b>	<b>Water Content</b>	<b>Additive</b>
Gasohol	1	0%	0 %
Gasohol	2	5%	0 %
Gasohol	3	5%	5 %
Gasohol	4	5%	4.5%
Gasohol	5	5%	4 %
Gasohol	6	5%	3.5%
Gasohol	7	5%	3 %
Gasohol	8	5%	2.5%
Gasohol	9	5%	2 %
Gasohol	10	5%	1.5%
Gasohol	11	10%	1 %
Gasohol	12	10%	0.5%

**5 Example 10h - Diesel**

<b>Fuel</b>	<b>No</b>	<b>Water Content</b>	<b>Additive</b>
Diesel	1	0%	0 %
Diesel	2	5%	0 %
Diesel	3	5%	5 %
Diesel	4	5%	4.5%
Diesel	5	5%	4 %
Diesel	6	5%	3.5%
Diesel	7	5%	3 %
Diesel	8	5%	2.5%
Diesel	9	5%	2 %
Diesel	10	5%	1.5%
Diesel	11	10%	1 %
Diesel	12	10%	0.5%

**Example 10i - Alternative Gasoline**

Consisting of alcohol and a blend of hydro carbons the majority percentage being alcohol

5

<b>Fuel</b>	<b>No</b>	<b>Water Content</b>	<b>Additive</b>
Alt Gas	1	0%	0 %
Alt Gas	2	5%	0 %
Alt Gas	3	5%	5 %
Alt Gas	4	5%	4.5%
Alt Gas	5	5%	4 %
Alt Gas	6	5%	3.5%
Alt Gas	7	5%	3 %
Alt Gas	8	5%	2.5%
Alt Gas	9	5%	2 %
Alt Gas	10	5%	1.5%
Alt Gas	11	10%	1 %
Alt Gas	12	10%	0.5%

**Example 11**

To record the visual aspects of phase separation for the one percent water and 0.1 percent titrations it was decided to scale up the volumes tenfold to enable accurate readings, therefore 1 litre of each fuel was transferred to each of twelve 2 litre measuring cylinders.

After the introduction of each titration the solution was gently stirred for twenty seconds. The resultant effect was left for ten minutes to settle before visible results were recorded.

**Example 11a**

<b>Fuel</b>	<b>No</b>	<b>Water Content</b>	<b>Additive</b>
Gasoline	1	0%	0 %
Gasoline	2	1%	0 %
Gasoline	3	1%	1 %
Gasoline	4	1%	0.9%
Gasoline	5	1%	0.8%
Gasoline	6	1%	0.7%
Gasoline	7	1%	0.6%
Gasoline	8	1%	0.5%
Gasoline	9	1%	0.4%
Gasoline	10	1%	0.3%
Gasoline	11	1%	0.2%
Gasoline	12	1%	0.1%

**Example 11b**



<b>Fuel</b>	<b>No</b>	<b>Water Content</b>	<b>Additive</b>
Gasohol	1	0%	0 %
Gasohol	2	1%	0 %
Gasohol	3	1%	1 %
Gasohol	4	1%	0.9%
Gasohol	5	1%	0.8%
Gasohol	6	1%	0.7%
Gasohol	7	1%	0.6%
Gasohol	8	1%	0.5%
Gasohol	9	1%	0.4%
Gasohol	10	1%	0.3%
Gasohol	11	1%	0.2%
Gasohol	12	1%	0.1%

**Example 11c - Diesel**

<b>Fuel</b>	<b>No</b>	<b>Water Content</b>	<b>Additive</b>
Diesel	1	0%	0 %
Diesel	2	1%	0 %
Diesel	3	1%	1 %
Diesel	4	1%	0.9%
Diesel	5	1%	0.8%
Diesel	6	1%	0.7%
Diesel	7	1%	0.6%
Diesel	8	1%	0.5%
Diesel	9	1%	0.4%
Diesel	10	1%	0.3%
Diesel	11	1%	0.2%
Diesel	12	1%	0.1%

**Example 11d - Alternative Gasoline**

Consisting of alcohol and a blend of hydro carbons the majority percentage being alcohol

<b>Fuel</b>	<b>No</b>	<b>Water Content</b>	<b>Additive</b>
Diesel	1	0%	0 %
Diesel	2	1%	0 %
Diesel	3	1%	1 %
Diesel	4	1%	0.9%
Diesel	5	1%	0.8%
Diesel	6	1%	0.7%
Diesel	7	1%	0.6%
Diesel	8	1%	0.5%
Diesel	9	1%	0.4%
Diesel	10	1%	0.3%
Diesel	11	1%	0.2%
Diesel	12	1%	0.1%

5

**Example 12****Exhaust Emissions**

The vehicle used was a 1993 California certified Mercury Cougar with 26,333 Miles on the odometer. This vehicle is equipped with a 3.8 litre engine with an SFI fuel system and has an inertia weight of 38,875 lbs.

10

A chassis dynamometer similar to a Clayton Water Break model was used in accordance with Federal Test Procedure CFR40 also known as the LA4 test.

15

Firstly the vehicle was pre-conditioned with indoline with the following sequence:

- 1/ Drain and fill the tank to 40% capacity with indoline.
- 2/ Disconnect the vehicle's battery to eliminate any mis-reading by a fuel computer.

3/ Drive vehicle for a period of 10 miles on the dynamometer in the **specified controlled conditions** and allow to soak for a minimum of 12 hours to a maximum, of 24 hours.

5 **Specified control conditions**

The test of additised fuel against base fuel was run with base fuel first.

The soak time from pre-condition to test was 15 hours, the soak temperature was 76° F and the barometer H.g. was 29.85.

10 The tests consisted of cold starts and test drives.

**Example 13**

**Emission Tests**

15

Emission tests were carried out on three fuel compositions containing different percentage blends of ethanol stabilised into a clear homogeneous fuel using Bi07.

The procedures are those in VTT Research Report NO. ENE824106/98.

20

The results of the test were compared with results obtained from a similar test carried out by a AB Svensk Biprovnng Motocenter (Swedish Engine and MOT test centre) on several fuel compositions including Swedish Mk1 diesel, generally regarded as the cleanest diesel available in Europe.

25

The comparisons gave evidence to a dramatic reduction in all measured emissions including CO<sub>2</sub>, -NO<sub>x</sub> and particulates.

**Test equipment and procedures**

30

All equipment used for exhaust dilution and collection, as well as concentration analysis of the gaseous regulated emissions, conform with the specifications of the Amendment 91/441/EEC of Directive 70/220/EEC.

- 5 A DC type chassis dynamometer manufactured by Froude Consine and an emission measurement system by Pierburg GmbH (FRG) were used.

Tests were conducted at normal test temperature ( $=23^{\circ}\text{C}$ ). The vehicle was preconditioned with running three times the EUDC part of the test, and soaked at the  
10 test temperature for 12 to 16 hours before the test.

The chassis dynamometer settings used for vehicle are presented in Table 5.

Table 5: Chassis dynamometer settings

Inertia	1360kg
$F_0$	7
$F_1$	0
$F_2$	0.046

15

The gaseous regulated emissions were divided into three sub-cycles. The first part included the first two individual sub-cycles of urban cycle, ECE15 (marked as Phase 1), the second phase was the rest of the ECE15 cycle (marked as Phase 2), and the third part was the extra urban portion (marked as Phase 3) of the current European  
20 test cycle (marked as 91/441/EEC).

#### Example 14

Different blends were made and the ratio of hydrocarbon: ethanol was measured by  
25 gas chromatography. The results were used to indicate the extent to which the content of the ethanol could be incorporated without exceeding an increase in the temperature at which the volatile ingredients evaporated. The results showed the amount of

ethanol that could be incorporated into the hydrocarbon without increasing the temperature at which the volatile components evaporated.

**Example 15**

5

The RVP value was measured according to ASTM D5191 and is the mean value of results calculated according to CARB, EPA and ASTM methods. These results demonstrate a tendency of the additive to reduce the RVP value, and that by selecting appropriate proportions a fuel composition can be made to meet the requirements of local legislation.

10

**Example 16**

Various tests were conducted according to ASTM D-5191.

15

**Example 17**

Various tests were conducted according to the 1975 US Federal City Gasoline Test.

20

## CLAIMS

1. A fuel additive comprising an alkoxyated alcohol, an alkanolamide and an alkoxyated fatty acid characterised in that the alkoxyated alcohol has a molecular weight of greater than 281 and the fatty acid (without ethoxylation) is a C<sub>16</sub> to C<sub>30</sub> fatty acid.
2. A fuel additive according to Claim 1 characterised in that the degree of ethoxylation of the ethoxyated alcohol is from 1 to 20.
3. A fuel additive according to Claim 1 characterised in that the ethoxyated alcohol has a molecular weight of from 281 to 790.
4. A fuel additive according to Claim 3 characterised in that the ethoxyated alcohol has a molecular weight of from 305 to 790.
5. A fuel additive according to Claim 1 characterised in that the alcohol ethoxylate is a Neodol or a mixture of ethoxyated Neodols.
6. A fuel additive according to Claim 5 characterised in that the alcohol ethoxylate is selected from one or more of Neodol 91-6, Neodol 91-8, Neodol 1-9, Neodol 23-3, Neodol 23-5, Neodol 23-6.5, Neodol 25 and Neodol 45.
7. A fuel additive according to Claim 1 characterised in that the alcohol ethoxylate is selected from one or more of Neodol 91-6, Neodol 91-8, Neodol 1-3, Neodol 1-5, Neodol 1-7, Neodol 1-9, Neodol 23-3, Neodol 23-5, Neodol 23-6.5, Neodol 25-3, Neodol 25-7, Neodol 25-9, Neodol 25-12, Neodol 45-2.25, Neodol 45-7 and Neodol 45-13.
8. A fuel additive according to Claim 7 characterised in that the Neodol is ethoxyated Neodol 91-6.

9. A fuel additive according to Claim 7 characterised in that the Neodol is ethoxylated Neodol 1-3
10. A fuel additive according to Claim 7 characterised in that the Neodol is  
5 Neodol 1-5
11. A fuel additive according to Claim 7 characterised in that the Neodol is Neodol 1-7.
- 10 12. A fuel additive according to Claim 1 characterised in that the fatty acid is a C<sub>16</sub> to C<sub>20</sub> fatty acid, or an ester thereof.
13. A fuel additive according to Claim 12 characterised in that the fatty acid is a C<sub>16</sub> to C<sub>18</sub> fatty acid, or an ester thereof.  
15
14. A fuel additive according to Claim 13 characterised in that the fatty acid is a C<sub>18</sub> fatty acid, or an ester thereof.
15. A fuel additive according to Claim 14 characterised in that the fatty acid is  
20 oleic acid, or an ester thereof.
16. A fuel additive according to Claim 1 characterised in that the alkoxyated fatty acid is an ethoxylated fatty acid, or an ester thereof.
- 25 17. A fuel additive according to Claim 16 characterised in that the fatty acid ester ethoxylate is an alkyl ester.
18. A fuel additive according to Claim 17 characterised in that the fatty acid is a C<sub>1</sub> to C<sub>10</sub> alkyl oleate.  
30

19. A fuel additive according to Claim 1 characterised in that the ethoxylated fatty acid is a polyoxyethylene-7-oleate oleate.
20. A fuel additive according to Claim 1 characterised in that the alkanolamide is a diethanolamide.
21. A fuel additive according to Claim 20 characterised in that the diethanolamide is a super diethanolamide.
22. A fuel additive according to Claim 1 characterised in that the additive comprises 20-80% v/v ethoxylated alcohol, 10-40% v/v diethanolamide and 10-40% v/v ethoxylated fatty acid, or an ester thereof.
23. A fuel additive according to Claim 1 characterised in that the additive comprises 30-70% v/v ethoxylated alcohol, 15-35% v/v diethanolamide and 15-35% v/v ethoxylated fatty acid or an ester thereof.
24. A fuel additive according to Claim 1 characterised in that the additive comprises 25% v/v of an ethoxylated fatty acid, or an ester thereof.
25. A fuel additive according to Claim 1 characterised in that the additive comprises 50% v/v of an ethoxylated alcohol.
26. A fuel additive according to Claim 17 characterised in that the additive comprises about 25% v/v of diethanolamide, 25% v/v of ethoxylated fatty acid, or an ester thereof and 50% v/v of ethoxylated alcohol.
27. A fuel composition comprising a hydrocarbon fuel and a fuel additive according to claim 1.



28. A fuel composition comprising diesel and a fuel additive comprising an alkanolamide, an ethoxylated fatty acid, or an ester thereof, and an ethoxylated alcohol, characterised in that the ethoxylated alcohol has a molecular weight of between 281 and 790.
- 5
29. A fuel composition according to any of Claims 27 or 28 characterised in that the additive to fuel ratio is 0.5 – 1:1200.
30. A fuel composition according to Claim 29 characterised in that the additive to  
10 fuel ratio is 1:1200,
31. A fuel composition according to Claim 30 characterised in that the additive to fuel ratio is 1:1000.
- 15 32. A fuel composition according to Claim 27 characterised in that the ethoxylated alcohol is Neodol 91/3.
33. A fuel composition according to Claim 27 characterised in that the fatty acid ethoxylate, or an ester thereof, is derived from a fatty acid having from 7 to 20  
20 carbon atoms.
34. A fuel composition according to Claim 33 characterised in that the fatty acid ethoxylate, or an ester thereof, is derived from a fatty acid having from 10 to 18  
25 carbon atoms.
35. A fuel composition according to any of Claims 27 or 28 characterised in that the fatty acid is a fatty acid ester
36. A fuel composition according to any of Claims 27 or 28 characterised in that  
30 the fatty acid ester is an alkyl C<sub>1</sub> to C<sub>10</sub> ester.

37. A fuel composition according to any of Claims 27 or 28 characterised in that the fatty acid is a polyoxyethylene-7- oleate derivative.
38. A fuel composition according to Claim 38 characterised in that the polyoxyethylen-7-oleate has as molecular weight of 500 or above.
39. A fuel composition according to Claim 28 characterised in that the mixture also comprises an oxygenator.
40. A fuel composition according to Claim 39 characterised in that the oxygenator is an alcohol.
41. A fuel composition according to Claim 40 characterised in that the alcohol is ethanol.
42. A fuel composition according to Claim 41 characterised in that the fuel comprises from 0 to 25% v/v ethanol.
43. A fuel composition according to Claim 42 characterised in that the fuel comprises from 1 to 10% v/v ethanol.
44. A fuel composition according to Claim 42 characterised in that the fuel comprises from 18 to 25% v/v ethanol.
45. A fuel composition according to Claim 27 characterised in that the fuel is gasoline and the additive to fuel ratio is 1:1000.
46. A fuel composition according to Claim 28 characterised in that the additive to fuel ratio is 1:500.

47. The use of an ethoxylated alcohol with a molecular weight of greater than 281 in the manufacture of a fuel additive according to Claim 1.

48. The use of an ethoxylated alcohol with a molecular weight of from 281 to 790  
5 in the manufacture of a fuel additive according to Claim 1.

49. The use of an ethoxylated alcohol with a molecular weight of greater than 281 in the manufacture of a fuel composition according to either of Claims 27 or 28.

10 50. A method of running an engine adapted to use an alcohol-based fuel, comprising adding to the fuel a miscible additive selected to solubilise the fuel and the additive so eliminating the deposit of by-products formed during the combustion of the fuel.

15 51. A fuel additive or a fuel composition substantially as described with reference to the accompanying examples.

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# INTERNATIONAL SEARCH REPORT

International application No

PCT/GB 00/01954

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C10L1/14 C10L1/10 C10L10/00 C10L10/02 C10L1/32

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C10L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

PAJ

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
E	WO 00 36055 A (WILLIAMSON IAN VERNON ;AAE HOLDINGS PLC (GB); HAZEL CLIFFORD JAMES) 22 June 2000 (2000-06-22)  claims 1-9,12,14,16-20	1-3,5, 12-14, 16,17, 20-27, 29-31, 33-36, 45-50
X	WO 99 20715 A (PURE ENERGY CORP) 29 April 1999 (1999-04-29) cited in the application example 2  --- -/-	28-31, 35,36,49

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/GB 00/01954

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 98 17745 A (WILLIAMSON IAN VERNON ;HAZEL CLIFFORD JAMES (GB)) 30 April 1998 (1998-04-30) cited in the application the whole document	28,49
A	GB 2 217 229 A (UNIV CITY ;ENERSOLVE CHEMICAL COMPANY LIM (GB)) 25 October 1989 (1989-10-25) cited in the application the whole document	1-51

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/GB 00/01954

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 0036055 A	22-06-2000	NONE	
WO 9920715 A	29-04-1999	US 6074445 A AU 9809798 A EP 1027410 A NO 20001972 A ZA 9809525 A	13-06-2000 10-05-1999 16-08-2000 08-06-2000 20-04-1999
WO 9817745 A	30-04-1998	AU 4710097 A DE 19782068 T GB 2334964 A, B SE 9901364 A	15-05-1998 11-11-1999 08-09-1999 16-04-1999
GB 2217229 A	25-10-1989	NONE	